A LABORATORY MANUAL

OF

EXPERIMENTS IN

GENERAL CHEMISTRY

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Before entering upon their work, the pupils are to carefully read the general laboratory instructions on page 469 of Freer's Text Book of General Descriptive Chemistry. Full notes of the results of all experiments are to be kept and all the questions asked in the Manual are to be answered; where additional instructions are needed, the teacher can be consulted.

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LABORATORY MANUAL

OF

EXPERIMENTS IN CHEMISTRY.

OXYGEN.

1. Heat 3 grams of mercuric oxide in a hard glass tube and test for oxygen with a glowing pine splinter.(?) What distills upon the sides of the tube? [Equations are to be written for all reactions.]

2. Heat about 15 grams of manganese dioxide in an iron tube in a combustion furnace and collect some of the evolved gas over water in a pneumatic trough. Test the gas.(?) A safety bottle should be placed between the furnace and the trough.(?) (Note 3.)*

3. Heat 2 or 3 grams of potassium chlorate in an air bath at 100° C. for 15 minutes.(?) Weigh off about one gram in a weighed porcelain crucible and heat with free flame, at first gently (?) until the mass fuses and again becomes solid.(?) Cool in a dessicator and weigh, noting loss in weight. Heat again with free flame until no more gas escapes. Cool and weigh.(?) What remains? Test an aqueous solution of the residue with silver nitrate solution.(?) Test a solution of potassium chlorate with silver nitrate.(?) What has heat effected? From your data calculate the per cent. of oxygen in potassium chlorate and com-

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* The Notes referred to are the Laboratory Notes in the Appendix of Freer's Descriptive Chemistry,

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pare it with the per cent. deduced from the atomic weights.

4. Heat potassium chlorate in a hard-glass test tube. When the salt melts remove tube from flame and add to the melted mass a little finely powdered manganese dioxide.(?) Test the gas.

5. Prepare an oxygen generating flask. (Fig. 2.) (Page 19.) Mix in a mortar 3 gms. of manganese dioxide and 15 gms. potassium chlorate, (?) place in flask, insert the stopper and tube and heat gently, collecting all except the first few hundred cubic centimeters in flasks over a pneumatic trough. What volume of gas is obtained? What weight? (Note 5.)

6. Into one of the flasks insert a glowing pine splinter.(?) Into your flasks insert in deflagrating spoons (Fig. 3, page 23) (a) A piece of charcoal. (b) A little sulphur. (c) A piece of phosphorus as large as a pea. (Care!!) Note changes if any. Now introduce a, b, c, as above, heating first in the Bunsen flame.(?) Do the contents of the flasks support combustion after the reactions have taken place? Notice odor of (b). (?) Note 6. Shake some litmus solution with b and c. (?) Tip a piece of steel wire with melted sulphur, ignite the sulphur and introduce into a jar of oxygen. (?) (Note 7.) What is combustion? Kindling temperature? Difference between combustion in air and in oxygen?

HYDROGEN.

7. Proceed as in Note 8. (Fig. 6 Page 33.)

8. Prepare generating flask (Fig. 7, Page 37.) Place 5 gms. of zinc in flask and pour gradually through the funnel tube, dilute sulphuric acid (1 part strong sulphuric acid to 6 parts water, Note 12) and collect all the gas.

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* References are to Freer's Descriptive Chemistry.

Note amount of gas. What weight of hydrogen does it represent? What volume and weight of hydrogen is evolved by the solution of one gm. of pure zinc in sulphuric acid?

9. Reject contents of first flask after measuring. Wrap a towel around second flask (?) and bring its mouth near a flame? Insert a burning splinter into the last flask held mouth downward? Is hydrogen combustible? Does it support combustion?

10. For method of purification of hydrogen see Note 13.

11. Recharge your apparatus using 10 gms. of zinc, and proceed as directed in Notes 17 and 18.

12. After action is over, filter the contents of generating flask, evaporate to saturation (?) and set aside to cool (?) How much zinc sulphate may be produced from 25 gms. of zinc? What weight of sulphuric acid does it represent?

13. Pour hydrogen upward from one test tube to another, testing contents of upper tube.

14. Pass hydrogen through a U tube filled with fragments of potassium hydrate and connected with an ordinary clay pipe by means of a rubber tube, and show that hydrogen is lighter than air by producing soap bubbles which rise.

15. Show by means of diffusion apparatus (Note 15 Fig. 12 Page 55) that hydrogen readily passes through porous substances.

16. Pass steam through an iron tube containing iron filings, and heated to redness in a combustion furnace (?). Test the gas. A safety-bottle (?) should be placed between

the furnace and the pneumatic trough. What metals decompose water at ordinary temperatures? What connection between metallic nature and readiness of decomposition of water? What is hydroxyle? Write formulæ of a number of hydroxides not given in the text. Resemblance between action of sodium on water and that of zinc on sulphuric acid.

WATER.

17. Arrange an apparatus as shown in Fig. 8, Note 21. The generator (Fig. 9), (page 44), contains zinc and dilute sulphuric acid. The wash-bottles (Fig. 8, page 41) are filled as in note 21. Proceed as in note 21. After the hydrogen has been passing two or three minutes (?) heat the copper oxide with the bunsen flame and pass a slow stream of hydrogen for 15 minutes. Note color change (?). Now drive all traces of moisture from the copper oxide tube into the weighed U tube, (Fig. 14, page 63) and allow the apparatus to cool in a stream of hydrogen (?). Detach and weigh the tubes a second time after expelling hydrogen by a current of dry air. Calculate from your data the composition of water by weight.

18. Procure an apparatus as in Fig. 4, page 27. To the ends of the two insulated copper wires, provided with platinum electrodes, connect two or more bichromate cells. Fill the glass vessel, and also the tubes, with distilled water and turn on the current. Why is there no action? Now add to the water about one-tenth its volume of concentrated sulphuric acid (?). What gas collects at the anode? At the kathode? When sufficient gas has collected, remove the plates from the liquid and carefully measure the volumes of gases. Test each gas. Is there exactly two

volumes of hydrogen to one of oxygen? If not why not?

19. Fill a small test tube two-thirds full of hydrogen, then fill it up with oxygen. Wrap a towel around it and bring its mouth to a flame (?) Care!

20. Heat in test tubes (a) some crystals of sodium sulphate which have not lost their luster (?), (b) zinc sulphate (?), (c) copper sulphate (?).

21. Weigh out about 2 gms. of copper sulphate crystals in a weighed porcelain crucible and heat at 100° in an air bath for one hour. Cool in a dessicator and weigh (?). Heat again at 200° for one hour. Cool in dessicator and weigh (?). Change in appearance? In weight? Per cent. of loss in each case? Explain.

22. Dissolve anhydrous copper sulphate in water. Note change in temperature (?). Dissolve sodium nitrate in water (?) What is water of crystallization? Do all crystals contain it? What is deliquescence? Efflorescence?

23. Dissolve about one gm. of copper sulphate in 100 c. c. of water. Place in a flask supported on a retort stand, attach a condenser, with receiver, and gently heat the solution until nearly all of it has collected in the receiver. Test the contents of flask and receiver with blue litmus paper (?). What is solution? What is the difference between a chemical compound and a mechanical mixture? Make a hot, saturated solution of sodium chloride and allow it to cool? Examine the crystals under the microscope? How is the solubility of solids in liquids generally affected by heat? Of gases in liquids? How may water be freed from non volatile impurities?

HYDROFLUORIC ACID.

24. Cover one side of a glass plate with a thin coating of wax or paraffine, and cut any design, taking care to expose the glass. Place the glass, with the coated side downward over a lead trough containing a mixture of calcium fluoride and concentrated sulphuric acid (Hood,) After some hours exposure remove the coating from the glass (?)

CHLORINE.

(Perform experiments under a hood).

25. Prepare a chlorine generating flask using a thin walled flask of about 500 c. c. capacity. Mix in a mortar 50 gms. of manganese dioxide and 50 gms. of sodium chloride. Add 150 c. c. of sulphuric acid, one part water to two parts of concentrated acid. Heat gently in the flask, pass the gas through a bottle containing concentrated sulphuric acid (?) and collect it by displacement of air in dry flasks.

26. Introduce into these flasks:

(a) A little finely powdered antimony (?)

(b) A few pieces of heated copper foil (?)

(c) A piece of dry colored calico (?)

(d) A piece of moist colored calico (?)

(e) A piece of moist litmus paper (?)

(f) A piece of filter paper wet with oil of turpentine (?)

27. Make a saturated solution of chlorine in distilled water. Repeat 26 (d) and (e), with this solution.

Seal one end of glass a tube about one meter long and one centimeter in diameter. Fill it with strong chlorine water and invert it in a vessel containing the same solution. Place the tube in direct sunlight (?) What gas is collected? Test.

HYDROCHLORIC ACID.

28. Light a jet of hydrogen and introduce it into a jar of chlorine (?) (See also note 29.) Shake some blue litmus solution in the jar after chlorine color has disappeared (?) Do the same with a jar of chlorine (?) Under what conditions do hydrogen and chlorine unite (?)

29. Prepare apparatus as in 25, omitting wash bottle. Place in flask 15 gms. of sodium chloride and pour upon it 50 gms. dilute sulphuric acid (2 volumes sulphuric to 1 volume water). Collect two jars by displacement and the remainder in a beaker of water. Introduce into one jar a piece of moist blue litmus paper (?) and into the other a lighted taper (?) (Preserve your solution of sodium sulphate for Expt. 35.) 30. Proceed as in note 32.

Compare the solubility of chlorine and of hydrochloric acid in water.

31. To a little of your solution prepared in 29, add some iron filings in a test tube (?) Some zinc (?) Test the gas. Evaporate each solution in a porcelain dish (Hood,) and examine the product. Test a little of these solutions for chlorides (?) Dilute some of the solution of hydrochloric acid largely with water and taste (?)

32. To some dilute hydrochloric acid in a beaker containing a few drops of litmus solution, add, drop by drop, from a beaker, a solution of sodium hydrate. Note effect on color and taste of solution. When is a solution said to be neutral? Preserve the solution for Expt. 34.

33. Dissolve 3 gms. of caustic soda (weighed as accurately as possible on large scales) in 600 c. c. of distilled water, and preserve the solution in a stoppered bottle. Make a similar solution of caustic potash and bottle it. Now, find on side table sulphuric acid 1 pt. to 6 of water, nitric acid 1 to 1, and hydrochloric acid 1 to 1.

Of the first take 10 c. c. measured accurately and dilute it to 100 c. c. Of the latter solution take 20 c. c. (use pipette) and dilute to 100 c. c.

Into a clean beaker measure 10 c. c. of the last solution, add 4 or 5 drops of litmus solution, then add from a burette, caustic soda solution, a half c. c. at a time, till the litmus turns blue. Note number of c. c. (?). Now take again 10 c. c. of acid and a few drops of litmus solution and add caustic soda in a slow stream until one c. c. less than before has been added. Now add drop by drop, stirring with a glass rod till litmus just turns blue. Note the number of c. c. While the solution is red, it is acid, when it becomes blue, it is alkaline, at the turning point, it is neutral.

Repeat the experiment with 20 c. c. of acid. *Report results.*

Now repeat using caustic potash.

This being done, make solutions of hydrochloric and of nitric acid as directed for sulphuric acid, and proceed as with that acid.

Compare, by means of these data, the saturating power of the different acids. Also note the relative proportions of each acid required to neutralize equal weights of the two alkalies.

34. Evaporate to dryness the solution obtained in Expt. 32 and treat the substance thus obtained with a little concentrated sulphuric acid in a test tube (?).

35. Dissolve in water the residue left after preparation of hydrochloric acid, (Expt. 29), evaporate to saturation in a porcelain dish and set aside (?). Examine the crystals under the microscope (?). Dry some of these crystals between filter paper and heat them in a test tube (?). Make a solution of the crystals and add barium chloride solution (?). Add barium chloride solution to sodium chloride solution (?).

BROMINE.

36. Proceed according to note 33; using sulphuric, compare this reaction with that taking place in preparation of chlorine. Compare chlorine and bromine in regard to physical and chemical properties.

37. Make a solution of bromine in water and test its bleaching action as with chlorine (?).

38. Test the action of bromine on water in direct sunlight (?).

HYDROBROMIC ACID.

39. To a small piece of potassium bromide in a test tube add a few drops of concentrated sulphuric acid. Heat gently and insert a strip of moist blue litmus paper in the mouth of the tube (?). What gases are given off (?). Compare hydrochloric and hydrobromic acids as to stability (?).

40. Proceed as in Note 34.

41. Bring together, mouth to mouth, a jar of chlorine and one of hydrobromic acid. (?)

IODINE.

42. Mix 2 gms. of manganese dioxide and 4 gms. of potassium iodide, place in a 300 c.c. flask, add 5 c.c. of sulphuric acid and heat gently on a sand bath. (?)

43. Scrape some crystals from the neck of the flask with a glass rod and make the following tests:

(a) Examine under the microscope. (?)

(b) Try their solubility in water (?); in alcohol (?); in ether (?); in carbon disulphide (?); in an aqueous solution of potassium iodide. (?)

44. To a solution of iodine in water add a little starch paste. (Prepare starch paste by boiling a little starch in a test tube with water.)

45. Repeat Experiment 39, using potassium iodide. (?) Compare hydrobromic and hydriodic acids as to stability. (?)

46. To a dilute aqueous solution of potassium iodide, add, drop by drop, chlorine water. (?) Shake a portion with a little ether (?); with a few drops of carbon disulphide. (?)

47. Repeat (46), using bromine water instead of chlorine water. (?)

48. Repeat with potassium bromide and chlorine. (?) In the halogene group what change in the stability of the hydrogen compounds as the atomic weight increases? What change in the physical and chemical properties of the elements? What is the ratio of the specific gravity of the halhydric acids to each other?

SULPHUR.

49. Proceed as in Note 37.

50. Into a small retort or a test tube, bent as in Fig. 27, place a few grams of roll sulphur, heat gradually and

collect the distillate in a beaker of water. Notice the changes the sulphur undergoes while heating. Examine the sulphur in the beaker and allow some of it to stand. (?)

51. Proceed as in Note 39.

52. Occurrence of sulphur in nature? To what crystalline systems do the various forms belong? What is dimorphism? Polymorphism?

SULPHURETTED HYDROGEN.

53. Apparatus as in Experiment 8. To a few pieces of ferrous sulphide in the flask add dilute sulphuric acid. (?) Pass the gas through a small cylinder of water and collect some in water. . Solubility in water? Collect some in dry flasks by displacement. Specific gravity of the gas? Test the gas with lead acetate paper. (?) Test the combustibility of the gas. (?) See also Note 41.

54. Into a water solution of the gas pass chlorine.(?) Resemblance between this reaction and that between chlorine and water? What takes place when sulphuretted hydrogen is passed over heated iron? Water ditto? See also Note 40.

55. Pass sulphuretted hydrogen successively through solutions of salts of lead, tin, antimony, arsenic, mercury, copper and zinc. (?) Filter in each case and test the solubility of the precipitate in hydrochloric and in nitric acid. What metals thus give a precipitate with sulphuretted hydrogen? Why? Compare the sulphides with the oxides in regard to formula and solubility.

56. Measure out about 25 c. c. of potassium hydrate solution. Saturate one half with sulphuretted hydrogen

(?) then add the other half (?) Add a drop of this solution to a solution of lead acetate (?) Place a drop on a silver coin (?) What metals have soluble sulphides? Compare sulphhydrates with hydrates.

COMPOUNDS OF CHLORINE WITH OXYGEN AND WITH OXYGEN AND HYDROGEN.

Nomenclature and composition of these compounds?

57. To 30 grams of quicklime, add, gradually, 50 c. c. of water (?) (Difference between hydroxide and oxide? What is hydroxyle?) Place the mixture in a flask and pass chlorine till no more is absorbed (?) Add water, filter, add dilute sulphuric acid and try bleaching action of the solution on litmus and colored cloth. What would have been produced had you used sodium hydrate instead of slaked lime?

58. Make a solution of 10 grams of potassium hydroxide in 25 c. c. of water in a beaker, keep it warm and pass chlorine till saturated (?) Filter and allow to cool (?) Recrystallize from a little hot water (?) Examine the crystals under the microscope and compare them with those of potassium chlorate.

59. Heat some of the crystals on charcoal by means of the blowpipe (?) To some of the residue, in a test tube, add dilute sulphuric acid (?) To another portion of the residue dissolved in water, add a drop of silver nitrate solution (?)

Define precipitate. What is the cause of precipitation?

60. To a *small* quantity of potassium chlorate in a test tube, add a little concentrated sulphuric acid (?)

(Care ! !) [Hold the mouth of the test tube away from the face.]

61. To some potassium chlorate in a test tube, add hydrochloric acid and warm gently (?) Test the evolved gas (?)

How would you prepare potassium perchlorate? Perchloric acid?

How do the oxyacids of chlorine, illustrate the law of multiple proportions?

How does the stability of the free acids vary with the amount of oxygen in the molecule?

How does chlorine oxidize? Give cases of oxidation which you have had. Of reduction. Show connection between oxidation and reduction.

62. To a solution of potassium hydrate, add bromine water (?)

63. Repeat 62, using iodine instead of bromine (?)

COMPOUNDS OF SULPHUR WITH OXYGEN AND WITH OXYGEN AND HYDROGEN.

64. Where have we prepared sulphur dioxide? Apparatus as in Expt. 25. Proceed as in note 42. Sulphur dioxide may also be prepared by reduction (?) of sulphuric acid with charcoal, sulphur, silver, mercury, etc. Write equations for these reactions. What differences between bleaching by chlorine and that by sulphur dioxide? What is the valence of sulphur in sulphur dioxide? In sulphurous acid? In sulphuric acid?

65. Into a solution of sulphur dioxide, pass sulphuretted hydrogen (?)

Define anhydride. What is the anhydride of nitric acid? Of chloric acid? How is sulphur dioxide prepared commercially? Its uses? Does sulphurous acid oxidize in the air?

Enumerate the reactions in which you have used sulphuric acid. What is sulphuric anhydride?

66. Place a piece of wood in a test tube of concentratrated sulphuric acid (?)

67. Preparation of sulphuric acid. Proceed as in note 44.

68. Pour some sulphuric acid upon a little sugar (?)

69. To 50 c. c. of water in a beaker, add gradually 25 c. c. of concentrated sulphuric acid, stirring well. Note heat changes (?)

70. To potassium sulphate add a little concentrated sulphuric acid and heat. On cooling the solution, what separates? Sulphuric acid is dibasic. Why? Define primary salt. Secondary salt. Neutral salt.

71. To a dilute solution of potassium sulphate add successively—strontium chloride (?) barium chloride (?) lead acetate solution (?) Try the solubility of the precipitates in nitric acid.

What are the members of the sulphur family? Compare them as to physical and chemical properties.

Occurrence of sulphates in nature?

NITROGEN AND THE ATMOSPHERE.

75. Place a piece of phosphorus as large as a pea in a crucible cover and float this on a large cork in a pneumatic trough. (Fig. 33). Ignite the phosphorus with a hot glass rod and cover it with a beaker or a wide mouthed bottle. Allow it to stand until the water no longer rises in the vessel (?) What proportion of the air has disap-

peared? What remains? Test the residual gas with a lighted taper (?) What has become of the phosphorus? Test the water in the trough with litmus paper(?) Note 45.

73. Place a graduated gas tube mouth downward in a cylinder of water, adjusting it so that the water within and without the tube stands at the same level (?) (Fig. 34). Reduce the volume of air to standard conditions, (Page 167). Place a small piece of phosphorus on the end of a wire (care!!) and insert it in the tube so that the phosphorus is about one-fourth of an inch above the water. When the water has risen and covered the phosphorus, raise it again. Allow the apparatus to stand until the water no longer rises (?), adjust the levels as before, reduce to standard conditions and calculate the per cent. of oxygen in the air. Properties of nitrogen? Usefulness in the atmosphere? Evidences that air is a mechanical mixture, not a chemical compound.

74. By means of an aspirator, draw air slowly through two bottles containing respectively, sodium hydrate solution (?) and concentrated sulphuric acid (?) then through a hard glass tube filled with copper filings heated to redness. Collect and test the gas which passes over the copper (?)

75. Expose some clear lime water and baryta water to the air (?)

76. Aspirate air through two wash bottles containing sodium hydrate solution, then through an empty bottle. After a time remove the latter from the apparatus, pour in a little clear lime water, cork tightly and shake (?) Now burn a small piece of charcoal in the bottle. Cork and

shake (?) Necessity of carbon dioxide in the atmosphere? Quantity? Sources? Causes of removal? Other impurities in the atmosphere?

AMMONIA.

77. Arrange an apparatus as in preparation of hydrochloric acid. Place in the flask a mixture of 25 grams of ammonium chloride and 50 grams of lime, add sufficient water to cause the mixture to form lumps and heat gently on a sand bath. Collect the gas in a beaker of water (Hood). When the water is well saturated, fill several flasks by displacement of air (?). Specific gravity of ammonia compared with air (?). Compared with hydrogen (?).

78. Place one of your flasks, containing ammonia, mouth downward in water (?). Solubility of ammonia in water (?). Is ammonia combustible? Does it support combustion?

79. Heat gently, in a test tube, a small quantity of strong ammonia water, and pass a stream of oxygen through it. After a few seconds bring a flame to the mouth of the test tube (?) Can ammonia be easily liquified?

80. Place a dilute solution of ammonia in an evaporating dish and add, slowly, from a burette, dilute hydrochloric acid until the solution is neutral (?). Evaporate to saturation on the water-bath and set aside. Examine the crystals under the microscope.

81. Repeat, using sulphuric acid (?). Nitric acid (?). What are the alkalis? How does ammonia resemble them? What are ammonium salts? What are the sources of ammonia? How is it manufactured? Compo-

sition of ammonia by weight? By volume? Occurrence in nature?

COMPOUNDS OF NITROGEN WITH OXYGEN AND WITH OXYGEN AND HYDROGEN.

Learn nomenclature and composition of these compounds.

82. Proceed as in Notes 52, 53, 54 and 55.

83. Fuse a little potassium nitrate in a test tube and add a small piece of sulphur (?). (Care!!). Principle of gunpowder? Formation of nitrates in nature?

84. Proceed as in Note 58.

85. Boil some potassium nitrate solution for ten minutes with zinc dust (?) Filter, add a drop of sulphuric acid and a drop or two of potassium permanganate solution (?). See Note 59 on decomposition of nitrates of the alkalies by heat.

86. Proceed as in Note 59.

PHOSPHORUS AND PHOSPHINE.

In what experiments have you already used phosphorus?

87. Bring together in a porcelain crucible a little phosphorus and iodine (?). Care!!)

88. Allow a little phosphorus to stand in the air. (?)

89. Demonstrate the low kindling temperature according to Note 60. (Second Part.)

Difference of activity of phosphorus and nitrogen. (?)

90. Place a little red phosphorus on the end of a pine stick and hold it in the flame of a bunsen burner. (?)

91. Compare the solubility of yellow and red phosphorus in carbon disulphide. (?) How is red phosphorus

prepared? How converted into the yellow variety? Compare yellow and red phosphorus as to physical and chemical properties.

92. For method of preparation of phosphine see Note 61. (Close the connection between the hydrogen generator and the flask before heating the caustic potash solution.) What causes spontaneous combustion? How does phosphine resemble ammonia? How does the preparation of phosphine from calcium phosphide resemble that of other hydrogen compounds of the nonmetals?

COMPOUNDS OF PHOSPHORUS WITH OXYGEN AND WITH OXYGEN AND HYDROGEN.

93. Proceed according to Note 62. What anhydrides have we met with thus far?

Compare the acids of nitrogen and of phosphorus. Look up ortho, meta and pyro-phosphates. Give the composition and properties of phosphorous acid. Occurrence and importance of the phosphates in nature.

ARSENIC.

Occurrence in nature? Appearance? Solubility in (a) hydrochloric (?), (b) nitric (?), (c) aqua regia (?). (Care!)

94. Proceed as in Note 64.

95. Heat a little arsenic on charcoal before the blowpipe? Notice the odor. (?)

96. Proceed as in Note 63. Compare arsine and ammonia in respect to stability. Is arsine basic?

COMPOUNDS OF ARSENIC WITH OXYGEN AND WITH OXYGEN AND HYDROGEN.

Use arsenic trioxide. Solubility in water? In hydrochloric acid? In nitric acid? In solution of caustic alkali? In sodium carbonate solution? The arsenites of the alkalis are derived from meta-arsenious acid ($H\,As\,O_2$) not yet isolated. The other arsenites from ortho-arsenious acid ($H_3\,As\,O_3$). What arsenites are soluble in water?

97. Into a solution of arsenic trioxide in hydrochloric acid pass hydrogen sulphide (?). Filter. Try the solubility of the precipitate (a) in hydrochloric acid (?), (b) in nitric acid (?), (c) in aqua regia (?), (d) in caustic alkali (?), (e) in ammonium sulphide (?), in ammonium carbonate (?). Acidify the ammonium sulphide solution. Make a solution of arsenic trioxide in potassium hydrate and add a drop of iodine in potassium iodide (?).

98. Prepare some arsenic acid by treating arsenic trioxide with nitric acid. (Hood.) Evaporate to dryness and dissolve in water. Into some of this solution, acidified with hydrochloric acid and boiling hot, pass hydrogen sulphide (?). Do the same with a cold solution (?). What arsenates are soluble? Reducing agents change arsenic acid into arsenious acid.

ANTIMONY.

Occurrence in nature? Appearance? Solubility in hydrochloric, nitric and aqua regia. For the following experiments use a solution of antimony chloride in hydrochloric acid.

99. Add potassium hydrate, drop by drop, finally in excess.

100. Make a solution of antimony trichloride in a little water. To this add an excess of water (?). (Formation of basic salts?) Contrast basic with acid salts.

101. Treat a portion of your antimony trichloride solution as in Expt. 97.

102. Treat some antimony with concentrated nitric acid (Hood) and evaporate to dryness on the water bath (?). Try the solubility of the residue in water (?). In hydrochloric acid (?). In nitric acid (?). In potassium hydroxide solution (?).

103. Prepare a solution of antimonic acid in hydrochloric acid and pass hydrogen sulphide into it (?). Treat the precipitate as directed in Expt. 97.

104. Proceed as in Note 65.

Compare the elements of this group—nitrogen, phosphorus, arsenic and antimony, and note the change in properties as the atomic weight increases. Compare with the chlorine and the oxygen group.

CARBON.

Forms of carbon? Compare with those of sulphur. What is charcoal?

105. Proceed as in Note 67. Uses of animal charcoal in the sugar refinery?

106. Proceed as directed in Note 69.

107. See Note 66.

108. What is methane? Heat in a hard glass tube, sealed at one end, a mixture of equal parts of anhydrous sodium acetate and soda lime, and after the air has been expelled from the apparatus collect some of the gas over water. Is it combustible? Formation of methane in nature?

COMPOUNDS OF CARBON WITH OXYGEN.

109. Force the gases from the lungs through clear lime water (?). Add a drop of hydrochloric acid to the precipitate (?).

110. To a little sodium carbonate in test tubes add successively dilute acids, (a) Acetic (?), (b) Hydrochloric (?), (c) Nitric (?), (d) Sulphuric (?). In each case hold in the mouth of the test tube a glass rod, on the end of which is a drop of lime water (?).

111. Apparatus as in preparation of hydrogen. Proceed as in Notes 72 and 73.

112. Into a solution of caustic soda pass carbon dioxide to saturation. Evaporate to dryness. Heat to redness in a crucible. Is the substance still a carbonate? Name the important natural carbonates. What metals have soluble carbonates? Is carbonic acid monobasic or dibasic? What is the rule as to the stability of acids, the anhydrides of which are gases?

CARBON MONOXIDE.

113. Occurrence in stoves? What is water gas? Proceed as in Notes 70 and 71. (Care!! as the gas is intensely poisonous).

114. (a) Pass dry carbon dioxide slowly over carbon heated in a hard glass tube, wash the gas as in the previous experiment, collect and test.

114. (b) Pass carbon monoxide over copperoxide heated in a hard glass tube, and test the resulting gas with lime water (?). What evidence have you that carbon monoxide is a reducing agent?

THE FLAME.

What is the average composition of illuminating gas?

115. Proceed as directed in Note 69.

116. Take a piece of charcoal with a plane surface and make a depression by pressing a coin against it and turning it around several times. Into this depression place a mixture of 3 parts of sodium carbonate and 1 of lead oxide and heat in the reducing flame (?) of the blowpipe until fusion takes place.

The blowpipe is to be used with a small luminous flame of the Bunsen burner. Separate the fused mass, place it in a mortar, add some water and triturate. What has become of the lead oxide? Why use charcoal?

117. Repeat Expt. 116 using copper sulphate instead of lead oxide (?)

118. Repeat using ferric oxide (?) Identify by means of the magnet.

119. Repeat using calcium sulphate. Potassium sulphate. Take a small piece of the fused mass obtained from the sulphates, place it on a bright silver coin and moisten it with a drop of water (?) Where have you met with this reaction?

120. Heat a small piece of lead on charcoal in the oxydizing flame of the blowpipe (?)

121. Treat as in 120 a piece of zinc (?) A piece of antimony (?) Uses of carbon in the reduction of metals from their ores?

COMPOUNDS OF SILICON WITH OXYGEN AND WITH OXYGEN AND HYDROGEN.

Learn the properties of silicon and of its compound with hydrogen. For the following experiments use a solution of sodium silicate. Reaction of the solution toward litmus? Concentrate the dilute solution on the water bath (?) Solubility of sodium silicate in water? Solubility of other silicates?

122. To the dilute solution add hydrochloric acid and allow it to stand (?) (Very dilute solutions must be concentrated before the silicic acid separates.)

123. Add hydrochloric acid in excess to a solution of sodium silicate, evaporate to dryness, boil with water, filter, and dry the remaining silicic acid. Properties? Solubility in water? In acids?

124. Add hydrofluoric acid to a little silicic acid in a platinum crucible and evaporate several times (?) (Hood.)

125. Powder some glass and evaporate in a platinum crucible with hydrofluoric acid and a little sulphuric acid until the latter begins to pass off (?) What remains? Occurrence of silicon in nature? What are ortho silicates and meta silicates? How does silicon resemble carbon?

126. Proceed as directed in Note 74. (Part 2.)

TIN AND ITS COMPOUNDS.

Appearance of the metal?

127. Try the solubility of tin in hydrochloric acid. In cold, dilute nitric acid (?) In hot and concentrated nitric acid (?) For the following experiments use a freshly prepared solution of stannous chloride.

Add, drop by drop, finally in excess, a solution of caustic alkali (?) Boil the solution (?) Repeat, adding ammonia (?) Sodium carbonate (?)

128. Treat a solution as directed in Expt. 97.

129. To some stannous chloride solution add mercuric chloride (?) Ferric chloride (?) Potassium dichromate (?) What reducing agents have you studied thus far?

130. Prepare a solution of stannic chloride by passing chlorine through a warm solution of stannous chloride (?). To this add, gradually, finally in excess, caustic alkali (?). Solubility of stannic acid in hydrochloric acid?

131. Treat some tin with concentrated nitric acid (Hood) and evaporate to dryness on a water bath (?). Solubility of the residue in water (?), in hydrochloric acid (?), in nitric acid (?), in caustic alkali (?).

132. Into a solution of stannic chloride, free from chlorine,(?) pass hydrogen sulphide and treat the precipitate as directed in Expt. 97. Name the important alloys of tin. Of antimony? What elements that we have studied, act both as bases and as acids?

BORON AND ITS COMPOUNDS.

For these experiments use borax.

133. Dissolve some borax in four times its volume of boiling water, add hydrochloric acid and set aside (?). Test the solubility of boric acid crystals in water (?); in alcohol (?). Filter, press the crystals between filter paper, dissolve in alcohol and set fire to the solution (?). Try the same experiment with borax (?). Occurrence of borax and boracic acid in nature? Their uses? Valence of boron?

ALUMINIUM AND ITS COMPOUNDS.

Occurrence in nature? Name some of its important compounds. Solubility of the metal in hydrochloric, nitric, and sulphuric acids, and caustic alkali?

130. To a solution of aluminium chloride add, drop by drop, finally in excess, caustic alkali (?)

131. Place some aluminium foil in a hard glass tube provided with a condenser, pass a stream of chlorine gas and heat the metal strongly by means of the Bunsen burner (?) (Hood)

When the student has completed the work up to this point, other and more advanced work, such as the determination of vapor densities by the Dumas, Victor Meyer, and Hoffman methods, will be assigned by the instructor.

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